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Synthesis and Characterization of AFe_2O_4 (A=CA, Co, CU) Nano-Spinels: Application to Hydrogen Photochemical Production under Visible Light Irradiation

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Abstract : Hydrogen from renewable sources, such as solar, is referred to as green hydrogen. The splitting water process using semiconductors, such as photocatalysts, has attracted significant attention due to its potential application for solving the energy crisis and environmental pollution. Spinel ferrites of the MF_2O_4 type have shown broad interest in diverse energy conversion processes, including fuel cells and photo electrocatalytic water splitting. This work focuses on preparing nano-spinels based on iron AFe_2O_4 (A= Ca, Co, and Cu) as photocatalysts using the nitrate method. These materials were characterized both physically and optically and subsequently tested for hydrogen generation under visible light irradiation. Various techniques were used to investigate the properties of the materials, including TGA-DT, X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), UV-visible spectroscopy, Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX) and X-ray Photoelectron Spectroscopy (XPS) was also undertaken. XRD analysis confirmed the formation of pure phases at 850°C, with crystalline sizes of 31 nm for $CaFe_2O_4$, 27 nm for $CoFe_2O_4$, and 40 nm for $CuFe_2O_4$. The energy gaps, calculated from recorded diffuse reflection data, are 1.85 eV for $CaFe_2O_4$, 1.27 eV for $CoFe_2O_4$, and 1.64 eV for $CuFe_2O_4$. SEM micrographs showed homogeneous grains with uniform shapes and medium porosity in all samples. EDX elemental analysis determined the absence of any contaminating elements, highlighting the high purity of the prepared materials via the nitrate route. XPS spectra revealed the presence of Fe3+ and O in all samples. Additionally, XPS analysis revealed the presence of Ca^{2+}, Co^{2+}, and Cu^{2+} on the surface of $CaFe_2O_4$ and $CoFe_2O_4$ spinels, respectively. The photocatalytic activity was successfully evaluated by measuring H_2 evolution through the water-splitting process. The best performance was achieved with $CaFe_2O_4$ in a neutral medium (pH ~ 7), yielding 189 µmol at an optimal temperature of ~50°C. The highest hydrogen production rates for $CoFe_2O_4$ and $CuFe_2O_4$ were obtained at pH ~ 12 with release rates of 65 and 85 µmol, respectively, under visible light irradiation at the same optimal temperature. Various conditions were investigated including the pH of the solution, the hole sensors utilization and recyclability.

Keywords : hydrogen, MFe_2O_4, nitrate route, spinel ferrite
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